

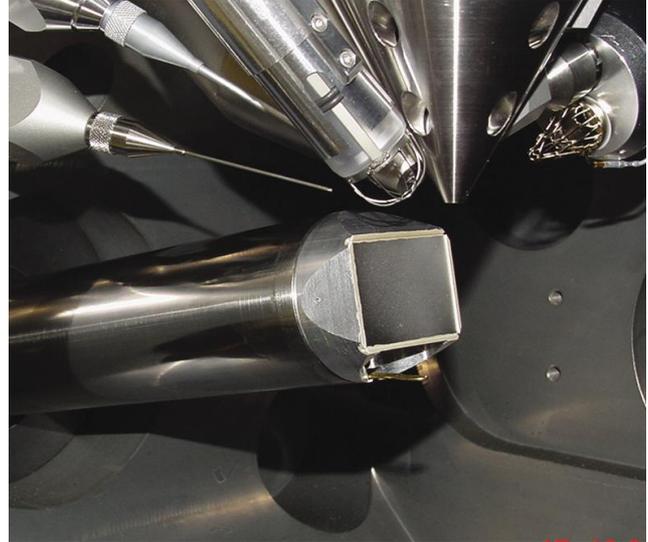
A look to the future: investigating the potential of the EBSD technique with combined focused ion beam (FIB) – SEM instruments

QUESTION:

What are the advantages of EBSD on combined FIB-SEM instruments?

Introduction

The recent emergence of integrated focused ion beam (FIB) instruments and scanning electron microscopes (SEMs) has caused considerable excitement in the materials and nanotechnology world. FIB-SEMs (commercially known as Dual-Beam or Cross-Beam instruments) allow the sectioning and shaping of samples on the nanometre scale, with integrated high resolution imaging. The potential of this technology, with the continuing drive towards miniaturisation and nano technology, is considerable, with applications in many fields. An added benefit for use with EBSD analyses is that the cut surfaces are ideal for obtaining diffraction patterns, with no further preparation necessary. Integrating EBSD technology into FIB-SEM instruments is not simple for 2 reasons. Firstly, the cross-over point of the electron and ion beams is at a very short working distance (5-8mm), and secondly the ion gun and associated gas injectors can obstruct the EBSD detector during insertion. However, Oxford Instruments EBSD HKLNordlys detector, combines a tapered nose design with specialised tilting interface plates to allow EBSD analyses at the electron-ion beam cross-over point (such as in the image above). This opens up a whole spectrum of new EBSD applications. In this application note we look at 3 examples of combined EBSD and FIB-SEM, illustrating the unique opportunities afforded by the sectioning capabilities of such instruments.



Analysis details

Example 1: Sectioning gold wires

FIB-SEM type: LEO 1540XB Cross Beam

EBSD System: HKL CHANNEL 5 with Nordlys Detector

	Sample 1	Sample 2
Grid dimensions:	251x228	261x232
Grid spacing:	100 nm	100 nm
Number of points:	57,228	60,552
Mapping speed:	5.5 / s	7.5 / s
Noise filtering level:	Low	Low

Examples 2 + 3: Textured ZnO Crystals, Sectioned W-filament

FIB-SEM type: FEI Strata 235 Dual Beam

EBSD System: HKL CHANNEL 5 with Nordlys Detector

	Example 2	Example 3
Grid dimensions:	251x110	83x264
Grid spacing:	50 nm	50 nm
Number of points:	14,530	21,912
Mapping speed:	7.5 pts / s	22 pts / s
Noise filtering level:	Medium	Low

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Results

Example 1: Texture variations in sectioned gold wires

Figure 1: Gold wires are commonly used to connect components on integrated circuits, and so it is essential that they have suitable microstructural characteristics (e.g. texture and grain size) in order to enhance their electrical properties. Analysing such wires using conventional techniques is, however, extremely difficult on account of their size – typically <math><25\ \mu\text{m}</math> in diameter. FIB instruments can easily section these wires, as illustrated in this secondary electron image, allowing subsequent EBSD analyses of the sectioned surfaces.

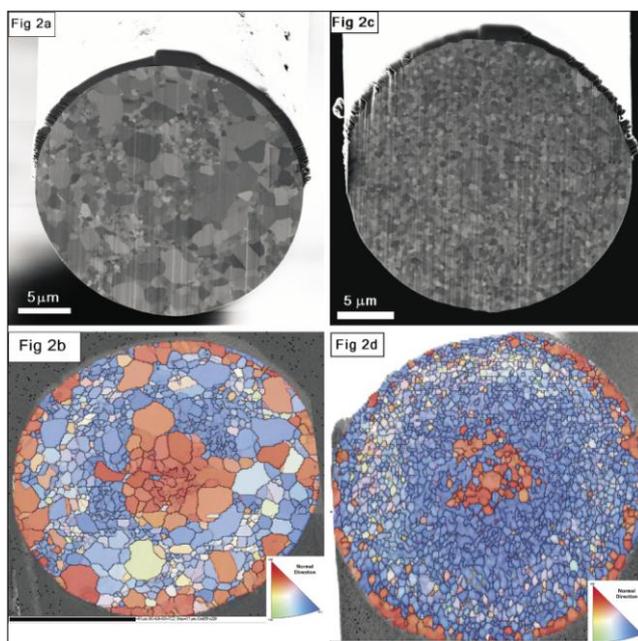
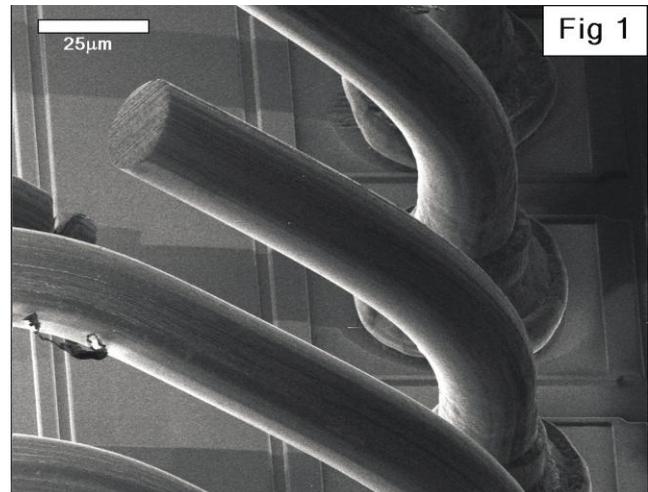


Figure 2: After sectioning, the wires need to be reoriented into a geometry suitable for forescatter imaging and EBSD analyses. Here the forescatter orientation contrast images and corresponding EBSD orientation maps are shown for 2 gold wires.

In the first sample (figures 2a and 2b), the grain size is relatively coarse and distinct textural domains can be seen. The centre and very edges of the wire are coloured predominantly red, indicating alignment of the $\langle 100 \rangle$ direction with the wire's long axis. The blue colour inbetween indicates alignment of the $\langle 111 \rangle$ with the long axis. The grain size in this section is $0.72\ \mu\text{m}$.

In the second sample (figures 2c and 2d), a similar texture is observed, but here the $\langle 111 \rangle$ fibre texture (blue) is much more dominant, with the $\langle 100 \rangle$ texture confined to the very centre and edges of the wire. The grain size in this section is also a lot smaller, with a mean of $0.41\ \mu\text{m}$.

Characterising these spatial and textural features on the sub-micron scale helps researchers to improve the processing of these wires, leading to improved performance in the final devices.



Example 2: Morphology and orientation of ZnO crystals

In this application, researchers are trying to grow ZnO crystals using precipitation from a liquid onto a glass substrate. Understanding both the morphology and the texture of these small crystals is important for refining the growth process. Figure 3 shows how the sectioning and subsequent EBSD analysis can easily provide the necessary information.

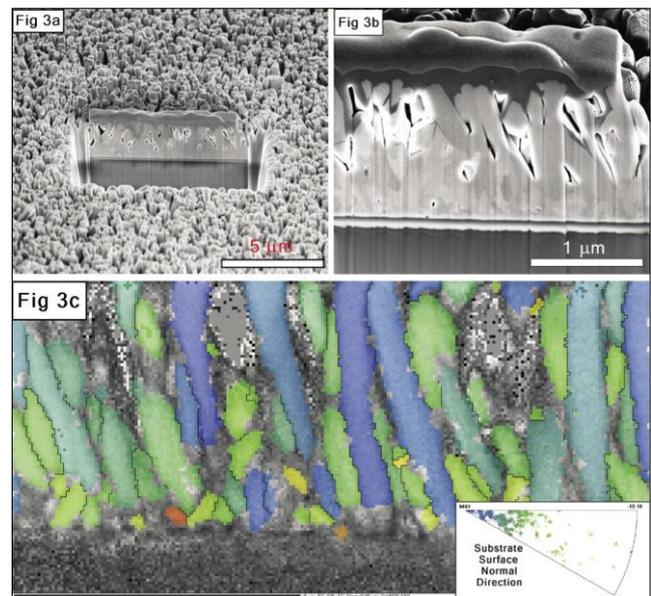


Figure 3: (a) Lower magnification secondary electron (SE) image of the ZnO surface showing the FIB cut and the crystal growth structure. (b) Higher magnification SE image of the FIB section. (c) EBSD orientation map of the same surface: the colours correspond to the misorientation from a basal plane orientation (see inset); most of the crystals have their $\langle 0001 \rangle$ axes aligned perpendicular to the substrate surface (grains shaded in blue colours), although there are a few more variable orientations closer to the substrate. The scale bar marks $5\ \mu\text{m}$.

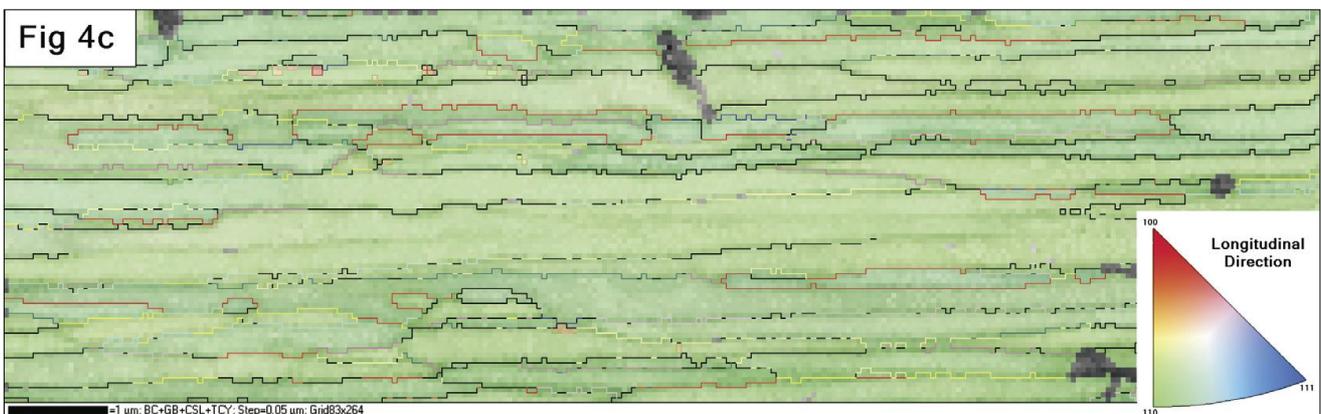
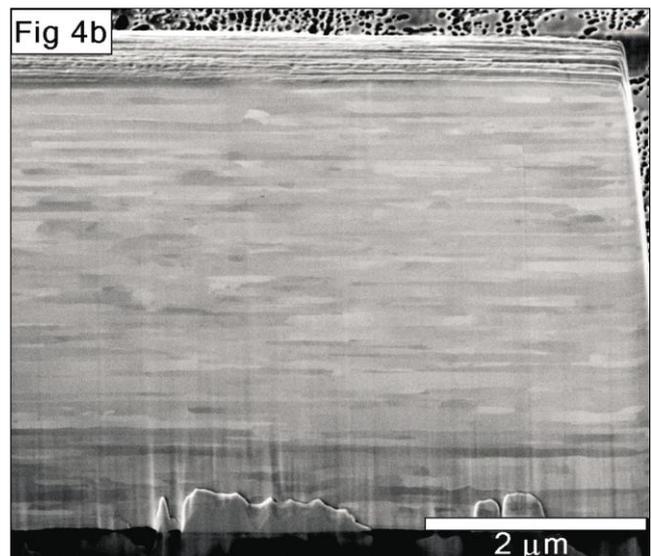
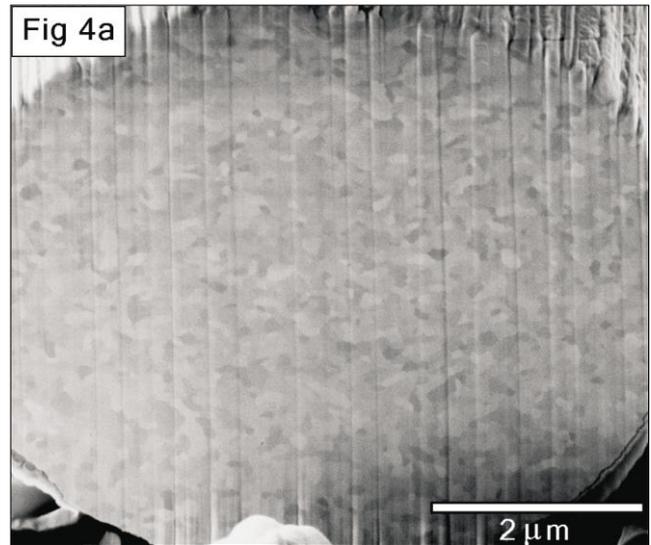
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Example 3: Textural variations in a tungsten filament

The performance of W-wires in lamp filaments is strongly dependent on the grain size, the grain shape and the texture. In this research, it was important to measure the microstructures of wires with diameters below $12\mu\text{m}$. Sectioning and preparing such delicate and fine samples would have been almost impossible with any other technique, but is straightforward with a FIB instrument. Figure 4 shows the sectioned surfaces and an example EBSD orientation map, clearly showing the highly textured nature of this sample.

Figure 4.

(a) SE image of the cross section cut surface. The fine grain structure is just visible, as are the vertical grooves caused by the FIB cutting process (b) SE image of a longitudinal cut surface, showing the very elongate nature of the grains. (c) EBSD orientation map of the longitudinal section. The colours correspond to the orientation of the wire's long axis in relation to the cubic crystallography (see legend – inset). The dominant green colour indicates that all the grains have their $\langle 110 \rangle$ direction aligned with the wire's long axis. The grain boundaries are shaded in black, with coincident site lattice boundaries (CSLs) in colours. It is clear that a large proportion of the boundaries are CSLs – this feature, coupled with the bamboo structure of the grains, will enhance the properties of the tungsten filament.



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Conclusions

Three brief examples of the application of the EBSD technique with combined FIB-SEM instruments have been shown. The potential for slicing and preparing surfaces on very small or difficult samples is clearly seen and this, coupled with the optimised EBSD system from Oxford Instruments, makes it a combination that will have a huge impact on materials science and nanotechnology in the coming years.

ANSWER:

FIB-SEM instruments provide the capability to section and prepare small, delicate or awkwardly shaped samples for immediate EBSD analysis, allowing the microstructural characterisation of samples that would otherwise be difficult or even impossible.

Acknowledgements

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